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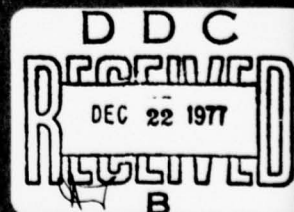
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PHOSPHONITRILIC FLUOROELASTOMER FUEL HOSE - UTILIZATION OF
EXTRUDED TUBES

Final Report - Contract DAAK70-76-C-0239

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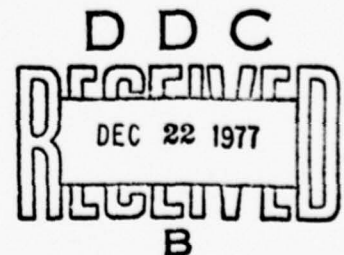
October, 1977

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Prepared for

U. S. Army Mobility Equipment
Research and Development Command

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SUMMARY

In the Arctic region, refueling operations must be conducted at temperatures as low as -70°F . Presently, there are no fuel resistant elastomers available that could be utilized for fuel hoses which would function at such temperatures. This work was part of a continuing effort to produce hose that could be utilized at -70°F .

An earlier contract effort (Contract No. DAAG53-75-C-0187) sponsored by the U.S. Army Mobility Equipment Research and Development Center (Ft. Belvoir, VA) showed that fuel hoses could be fabricated from a phosphonitrilic fluoroelastomer ($\text{PNF}^{\text{R}}\text{-LT}$). Laboratory tests indicated that the hose and rubber compounds of $\text{PNF}^{\text{R}}\text{-LT}$ were sufficiently flexible at -70°F .

The present investigation was an attempt to refine the manufacturing process for making fuel hoses of $\text{PNF}^{\text{R}}\text{-LT}$. The major refinement in the process was the utilization of extrusion for making tube sections of both the collapsible and suction type hoses.

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The initial phase of this work was to develop a PNF^R-LT compound which could be extruded and still maintained good low temperature properties. It was also desirable to maintain other properties such as fuel resistance, green strength, tear strength, tensile strength, and adhesion to rayon. The best balance of properties was achieved with an FEF black compound in which the optimum level of black was 33 P.H.R. In order to achieve good extrusions, it was also necessary to heat age the polymer at 300⁰F for 8.5 hrs. The resultant compound extruded very nicely and low temperature properties were good. Tensile strength and modulus were not up to specifications but probably as high as possible for any PNF^R-LT compound which can be extruded and possesses good low temperature flexibility.

Manufacturing of the collapsible and suction type hoses proceeded extremely well. Particularly satisfying was the extrusion process which produced excellent tube sections and greatly facilitated the entire production technique. There were some minor difficulties experienced in the calendering

of the very thin layer (0.014") for collapsible hose and with possible pull down of fabric into the tube section. These problems could probably be easily remedied by modifying the specifications on thicknesses of tube and layer sections.

Other refinements in the hose fabrication technique were also made. A reduction in fabric content was made which resulted in increased strike-through of rubber. Although adhesion data could not be obtained, it appeared that improved bonding of tube to cover was achieved. The reduction in fabric also produced a more flexible hose. Another modification which may have contributed to improved bonding of various sections of hose was the addition of a coupling agent to the cement formulation.

Despite the reduction in fabric content, both hoses showed good dimensional stability and physical strength. Hydrostatic pressure testing indicated that both hoses met all of the pressure test requirements. Inside diameters and weights were also within specifications.

Gehman test results indicated that the hoses should be flexible at -70°F . The reduction in amount of fabric in the hoses should enhance this flexibility. Resistance to Type II fluid was adequate, but volume increases in distilled water were above specifications.

Stress-strain data on samples removed from the hose showed tensile strength to be lower than specifications. Some lowering of tensile strength may have been caused by defects in the buffed samples of hose. Also, tensile strength was compromised in favor of extrudability and low temperature flexibility.

In general, results were quite satisfying and sufficient lengths of collapsible and suction hoses were made for field testing in the Arctic. This testing should provide valuable information on the performance capability of PNF^R-LT fuel hoses.

PREFACE

All investigations performed under Contract No. DAAK70-76-C-0239 are described in this report. The original contract was for a 6 month period from August 18, 1976 to February 18, 1977; subsequent modifications extended the contract to an 11 month period with work completed on July 18, 1977. The primary objective was to demonstrate that phosphonitrilic fluoroelastomer fuel hoses, capable of performing in Arctic environment, could be fabricated by utilization of an extrusion process for preparing tube sections.

This final report was prepared by the Central Research Laboratories of the Firestone Tire & Rubber Company. The work was sponsored and administered by the U.S. Army Mobility Equipment Research and Development Command, Ft. Belvoir, Virginia. Messrs. Philip Mitton and Charles Browne served as the Contracting Officer's Technical Representatives.

Special acknowledgement is given to Mr. Robert Lord and Mr. Al Turowsky for directing the fabrication of hoses at Boston Industrial Products Division of American Biltrite

Inc. Project management at Firestone was provided by Dr. J. A. Beckman, Manager of Elastomer Synthesis Division, and Dr. D. P. Tate, Assistant Director of Research. Many co-workers at the Firestone Central Research Laboratories assisted in the compounding and testing phases of this investigation, but special recognition is given Mr. J. F. Witner, Mr. E. K. Sanders, Mr. R. Sando and Dr. G. S. Kyker for their contributions.

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INTRODUCTION

The basic goal of this work was to develop phosphonitrilic fluoroelastomer (PNF^R-LT) compounds which could be extruded. Once developed, these compounds could be utilized in the production of PNF^R-LT fuel hoses which should be serviceable in extreme cold environments. The extrusion technique was to be used in production of the tube sections of these hoses. PNF^R-LT hoses should satisfy the Army's requirements for refueling operations in the Arctic.

The U.S. Army Mobility Equipment Research and Development Command has sponsored two earlier contract efforts directed toward preparation of PNF^R fuel hoses. The first investigation (Contract No. DAAK02-73-C-0464; 6/73-12/73) showed that fuel hoses could be fabricated from PNF^R-200 by a plied calendered sheet process. Hoses produced were not sufficiently flexible at -70°F. The second study (Contract No. DAAG53-75-C-0187) utilized a modified PNF^R, PNF^R-LT, which possesses improved low temperature flexibility. This effort resulted in hose with suitable low temperature flexibility and production

feasibility was again demonstrated for the "hand-built" technique (plied calendered sheets).

The preferred technique for producing both the collapsible and suction type fuel hoses is to extrude the tube sections and hand wrap only the cover sections. Thus, our objective for this contract was to develop compounds that extruded satisfactorily while still maintaining other desired properties (particularly low temperature flexibility). Once good extrusion compounds were developed, time and polymer permitting, we hoped to improve tear strength and adhesion to fabric. The best compounds developed would then be used in the manufacture of large lengths of collapsible and suction hoses.

INVESTIGATION

1. Polymer

In the initial compounding phase of this investigation, a blend of PNF^R-LT and PNF^R-200 was utilized for preliminary extrudability studies. This blend was identical to the blend used in a previous hose contract (DAA53-75-C-0187).

For the remainder of our compounding studies and for fabrication of final hoses, only the PNF^R-LT type polymer was utilized. It was unnecessary to add PNF-200 since the fluorine content of the PNF^R-LT was suitable to attain the desired balance in low temperature properties and fuel resistance. In order to process the PNF^R-LT, it was necessary to heat age the polymer for 8.5 hrs at 300°F. The heat aging reduced the "nerve" of the polymer and resulted in satisfactory extrusions.

In the previous contract, the fluorine contents of many of the polymers were < 40%. In this work, the PNF^R-LT's had fluorine contents of 44-49% and Tg's in the range of -73 to -81°C (most were -78.5 to -79.5).

2. General Approach

Previous studies with phosphonitrilic fluoroelastomers had shown that these polymers could be utilized to produce suction and discharge fuel hoses through use of plied calendered sheets. In manufacturing of hoses of this type, it is advantageous to extrude the tube sections. The basic objectives in this contract were to develop PNF^R-LT compounds which could be extruded satisfactorily and could be used to fabricate large lengths of discharge and suction hoses. While developing extrudable compounds, attention was always given to maintaining good low temperature flexibility and optimizing other properties (tensile strength, tear strength, adhesion to rayon, etc.).

3. Trial Hose Fabrication

Original plans for this contract effort did not call for any trial hose preparations. However, during the course of this work, a blistering problem arose in the testing of previously prepared hose. As a result, a trial fabrication of discharge hose was made in order to avoid the blistering problem in the hose prepared under the present contract.

4. Final Hose Production

The final hose building effort was an attempt to fabricate 125 feet of collapsible hose and 35 feet of suction hose with all of the tube sections extruded. The final compound chosen was basically that which could be extruded satisfactorily and maintained good low temperature flexibility.

5. Experimental Details

A. Instruments

1. Laboratory Rubber Mills

- a. 2" x 6", L. Albert and Son, Model A-6974, capacity: ca. 100 g of PNF^R-LT stock.
- b. 6" x 12", Farrel-Birmingham, Inc., Model 4467, capacity: ca. 2 lbs of PNF^R-LT stock.
- c. 10" x 12", Farrel-Birmingham, Inc., Model 44667, capacity: ca. 5 lbs of PNF^R-LT stock.

- 2. Brabender Mixer - Model PL-V150, C. W. Brabender (CWB) Instruments, Inc., capacity: ca. 120 g of PNF^R-LT stock. The CWB Type 100 extruder attachment was also used.

3. Banbury Mixer - Model B, Farrel-Birmingham, Inc., capacity: ca. 1900 g of PNF^R-LT stock.
4. Laboratory Balances
 - a. Sartorius, Model 2403 - used for weighing of curing agents and pigments for small batches (± 0.01 g).
 - b. Toledo, Model 3710 - used for weighing of pigments for large batches.
5. Instron Model No. 1130 - The Instron Corp., used for stress-strain measurements. The instrument was interfaced with an IBM 1130 Computer for computation of stress-strain data.
6. Shore Durometer - Shore Instrument and Mfg. Co., Inc.
7. Gehman Torsional Wire Apparatus - Wallace Test Equipment, Testing Machines, Inc., Amityville, NY.
8. Compression Set Jigs - 25% Deflection, Method B, were constructed at Firestone according to ASTM-D-395.

9. Forced Air Oven - Blue M Electric Co., for heat aging of polymer.
10. Cold Tension Recovery - The test instrument consisted of a measuring board to which are mounted several stretching devices consisting of a movable and a fixed clamp. Lines are engraved on the board at intervals corresponding to each 10% stretch, based on the length of the specimen between the 1/4 inch stubs.

B. Mixing Techniques

Brabender and Banbury Mixes --- A small amount of black is added to the mixer followed by addition of the polymer. The remaining black, MgO and stabilizer are added in increments. The compound is mixed for 8 to 10 minutes and dumped. Curing agent is then added to the masterbatch banded on a warm (130°F) mill.

C. Physical Test Methods

Test specimens were sheeted out on a rubber mill and press cured at 1000 psi unless otherwise stated. Tests were also run on specimens obtained from hose samples.

1. Stress-Strain -- ASTM-D-412, Die C, 73°F.

Specimens were cut from press-cured 1.5" x 4" x 0.040" or 6" x 6" x 0.075" slabs.

2. Shore "A" Hardness -- ASTM-D-2240, tests made on small cylinder (0.25" h x 0.53" d).

3. Compression Set -- ASTM-D-395, Method B, 25% Deflection, press cured cylinder. Low temperature tests -- ASTM-D-1229, same sample and conditions.

4. Gehman Low Temperature Measurements -- ASTM-D-1053.

Specimen 1.5" x 0.125" was cut either from a hose sample or a press cured 6" x 3" x 0.075" slab. An IBM 1130 Computer was programmed for computation and print-out of Gehman data and graphs. All testing was performed in accordance

with guidelines of Attachments #1 and #2 cited under Paragraph F. of Section F and Section J entitled "Special Provisions".

5. T-Adhesion Test -- A Firestone test performed as follows:

- a. Using a Hytronic Cutting Machine (Model A; United Shoe Machinery Corp.) and a 6" x 0.50" die prepare an adequate number of sheeted strips (0.110") for pad building.
- b. Ply one piece of rubber stock (6" x 0.50" x 0.110") onto one piece of calendered fabric backing (0.051").
- c. Place sample in building mold with fabric side down.
- d. Place ten cords (ca. 7" in length) with equal spacing on top of the two piece assembly.
- e. Invert another two piece assembly, made as in a. and b., on top of the cords so that cords are between two layers of stock to be tested.

- f. This assembly should now fit snugly into mold.
 - g. Cure adhesion pads as desired (usually 45' @ 320°F in this work).
 - h. Cords are pulled from the rubber pads by means of an 1130 Instron at a test speed of 10"/minute. The top grip is a special holder made for the cured sample, with a slot in the bottom to permit the sample to be inserted with the cord protruding. The bottom grip is a wedge type designed to exert increasing tightening as the cord is pulled.
 - i. The ten cords are pulled and averaged. Multiplication by two yields the lbs/in value reported.
6. Trouser Tear -- Test followed ASTM D1938 except for these modifications:
- a. Force necessary to propagate a tear measured on a rubber sheet (0.075") and not a plastic film.

b. The specimens consist of strips 3.5" x 2.0" with a longitudinal slit 2.5" long down the middle of the sheet.

7. Tension Recovery -- This test followed the procedure given in the Purchase Description of this contract and outlined as follows:

- a. With the specimens at a temperature ranging from 68°F to 78°F, they are clamped in the stretching devices and pulled back until the 1 1/2 inch portion of the specimen has been stretched to 100% elongation and fixed in that position.
- b. The stretching devices and the specimens shall be conditioned in a low temperature chamber for 166 hrs \pm 1 hr at -70°F \pm 2°F. The measuring board shall be conditioned for not less than two hours at the same temperature.
- c. With the test instrument and specimens still in the low temperature chamber, the movable clamp is released from its fixed position,

and the assembly is conditioned for an additional 30 minutes at -70°F.

- d. The final length of the specimen is determined 30 min. (\pm 10 sec.) after release of the clamps and with the stretching devices and specimens held at an angle of 15° from the vertical.
- e. The cold tension recovery percentage for each set of three specimens is calculated and averaged. The average value is used to determine compliance with the specification requirements.
- f. The percentage of cold tension recovery is computed from the formula:

$$\% \text{ cold tension recovery} = \frac{L_s - L_f}{L_s - L_o} \times 100$$

where: L_s = stretched length of specimen

L_f = final length of specimen

L_o = initial length of specimen

test specimens: 0.080" wide x 1.5" long
with 0.25" square at each
end.

8. Brittleness -- determined in accordance with ASTM designation D746.
9. Torsional Stiffness Ratio -- determined in accordance with Method 5612 of Federal Test Method Standard No. 601.
10. Existent Gum -- This test followed the procedure given in the Purchase Description of this contract and ASTM-D-381-70. A test sample of hose not less than 14 inches long is plugged with a clean corrosion resisting cylinder 2 inches long secured in place with a clamp. The sample of hose is filled to within 2 inches of the top with TT-S-735, Type II fluid. The top of the hose is then plugged in a manner similar to the bottom. The sample is stored in a vertical position for seven days at ambient temperature of 100°F (+2°F). Every 24 hours, the fluid is agitated for five minutes by moving the hose back and forth from vertical to horizontal positions at a rate of two cycles per

minute. At the end of seven days, the fuel is agitated again for five minutes and immediately removed. The fuel is tested for washed and unwashed existent gum in accordance with paragraphs 9.1-9.6 and 9.8-9.12 respectively of ASTM-D-381-70.

A modified version of this test utilizes diced samples of hose compound. A 5.0 g sample (< 70 mils thick) is cut from the hose and diced into approximately 1/16 inch squares and placed into a flask containing 250 ml of TT-S-735, Type II Fluid. The flask is kept for 48 hrs at 735°F (\pm 5°F) with occasional stirring. After filtration through Whatman 41H (or equivalent) paper, the existent gum content is determined (as above).

6. Preliminary Compounding Studies to Develop Extrudable Compounds

While waiting for completion of the preparation of the bulk of the polymer for this contract effort, we utilized

the same polymer blend (PNF^R-LT + PNF-200) used in the previous contract (DAAG53-75-C-0187) to do some preliminary extrusion studies. The first compound selected was, of course, the final compound used in the last hose construction. As clearly illustrated by the data in Table I, this 30 P.H.R. FEF formulation did not extrude satisfactorily. The surface of the extrudate was very poor with the lowest rating possible given for contour, corners, edge and surface.

Based on the first extrusion results, it was apparent that additional filler was required. In order to minimize losses in low temperature flexibility, large particle size silicas (Imsils) were added to FEF compounds. These formulations were evaluated in a Brabender extruder equipped with a 0.25" circular die. The switch to the Brabender extruder was made in order to conserve polymer and to speed up the extrusion screening process. Data in Table II indicate that only with 20 parts FEF and 30 parts Imsil could improvements in extrudability be realized. However, these improvements were marginal.

In Table III, the evaluations of some black compounds are illustrated. The 30 P.H.R. FEF formulation was checked again in order to assess the influence of higher extrusion temperature. Extrusion at 100°C produced no improvement. The addition of 20 P.H.R. of Austin black to this FEF compound gave only a slight improvement in extrudate surface (R203845). Increasing the FEF level to 40 P.H.R. (R203846) resulted in a significant improvement in extrusion. A fairly smooth surface was obtained with little die swell. However, as expected, the Gehman low temperature properties were adversely affected. The T₅ value was still respectable (-66°F), but the G value at -55°C was fairly high (771 psi). The final compound studied in this series was a 30 P.H.R. Shawinigan formulation that gave fair extrusions and good stress-strain and low temperature properties.

Both the FEF and Shawinigan blacks were evaluated at the 35 P.H.R. level (Table IV). The Shawinigan compound yielded good extrudates, while the FEF stock was only poor to fair in extrudability. Most of the other properties

were comparable, although the FEF formulation was slightly better in low temperature flexibility.

Besides good extrudability, other objectives of this work were to improve tear and tensile strengths. In an attempt to achieve these goals, the additions of a coupling agent (Silane A-174) and glass fiber to the 30 P.H.R. FEF stock were investigated. The Silane A-174 produced a modest improvement in tear strength, and the addition of a treated (coupling agent) fiber to the Silane stock (R203839) resulted in an additional increase in tear strength (Table V). Low temperature properties were good for both compounds. These results indicated that further studies with these formulations were warranted.

Again seeking improved tear strength, we evaluated an FEF compound containing Teflon-6 to which was added a silica and a coupling agent in an attempt to avoid delamination problems (evident in all Teflon stocks). The compound cured well as evidenced by stress-strain properties before and after extrusion (Table VI). The stocks could be delaminated but with greater difficulty than had been observed

with earlier Teflon-containing compounds. The gain in tear strength was very good, but low temperature properties and extrudability were poor.

Since Shawinigan black looked good, we investigated stocks with Shawinigan and Teflon 8-A combinations. Stress-strain properties and tear strengths were very good (Table III), but Gehman low temperature properties were poor. Also, delaminations were again possible.

7. Evaluation of the Large Lot of PNF^R-LT

In order to satisfy the requirements of this contract, over 200 lbs of PNF^R-LT had to be synthesized. This was done via 14 separate preparations in a pilot plant facility. The available properties of the resultant polymers are shown in Table VIII. Based on glass transition temperatures (T_g), all polymers were on spec for PNF^R-LT type products. A PNF^R-200, with 54% fluorine, will show a T_g of -68°C. Many of the batches were checked for %F, Gehman low temperature properties and % volume swell. Some of the polymers that were low in T_g and fluorine content showed high volume swells (>40%). However, these would be balanced out by

the polymers with somewhat higher Tg's and fluorine contents. Most of the polymers had Tg's in the desired range of -78.0 to -80.0°C. Thus, all fourteen batches were blended on a rubber mill to produce a uniform lot (K19166) of polymer for the remainder of this work.

8. Compounding Studies with Large Lot of PNF^R-LT (K19166)

Our first compounding effort with the large lot of polymer (K19166) was an evaluation of mill processing of this material in the standard 30 P.H.R. FEF formulation. The stock was very lacey and would not band on the mill, indicating that the polymer was too nervy and certainly would not extrude. Consequently, small samples of K19166 were heat aged for 4, 6, 8, 10 and 12.5 hrs at 300°F and then compounded (30 P.H.R. FEF). The results of studies on these heat-treated polymers are summarized in Table IX. Good mill processing was observed with all polymers. Extrusions improved with increased aging time, with the stock containing 12.5 hrs aged polymer showing good extrudates. The stress-strain properties seemed to fall off

slightly with increased aging time. Tear strength and Gehman properties seemed unaffected by heat aging time.

In view of earlier results, both FEF and Shawinigan blacks were evaluated at the 35 P.H.R. level with K19166 polymer that was aged for 8 hours at 300°F (Table X). Good extrusions were obtained for both compounds, with the Shawinigan formulation showing a slightly smoother surface. However, Gehman T₅ and G (-55°C) values were higher than desired, particularly for the Shawinigan compound.

Additional work was done with the FEF stock. First, a polymer that was aged for only 6 hrs @ 300°F was evaluated in the 35 P.H.R. formulation (Table XI). The desired increase in tensile strength (over R203891, Table X) was not realized. However, the extrusion was acceptable and low temperature properties were good. A comparison was also made of the 35 vs 33 P.H.R. FEF compounds with 7.5 hrs aged polymer. The processing and physical properties of these stocks were comparable, with the extrusions again being acceptable. The low temperature flexibility (Gehman test) was better for the 33 P.H.R. FEF

stock. The improved low temperature properties of the 35 P.H.R. FEF stock relative to the earlier examined compound (R203891, Table X) are unexplainable.

Earlier results with Silane A-174 (Table V) prompted a reinvestigation of this coupling agent with the new polymer. Using 33 P.H.R. FEF black, the Silane A-174 containing compound (R207709) again showed good normal properties. However, the tear strength and low temperature properties were not as good, and the extrusion seemed poorer with the coupling agent added (Table XII).

Also reexamined was the good tear compound that contained FEF and Quso WR-82 fillers along with Silane A-174 and Teflon-6. In this attempt, the Teflon-6 level was reduced from 3 to 1 P.H.R. in order to improve low temperature flexibility. The low temperature properties were improved, but insufficiently (Table XII). Also, tear strength was greatly reduced by the reduction in Teflon-6 level.

Glass fibers also gave earlier encouraging results, so compounds R207724 and R207725 were studied (Table XIII). It was found that reasonable properties could be attained,

but the extrudability was poorer with the glass fibers added. T-adhesions for these compounds to rayon were also very poor.

In another attempt to produce extrudable formulations with improved properties (particularly tear and tensile strengths), we examined a compound with an added co-agent (Saret 500) and a couple of formulations with SAF-Imsil filler combinations. The co-agent appeared to improve the extrudate surface but did not have much influence on tear or tensile strengths (Table XIV). Low temperature properties were poorer for the Saret-containing stock. Both of the SAF-Imsil compounds yielded rather poor extrusions and no improvements in tear or tensile strengths. Unfortunately, our control compound, containing 33 P.H.R. FEF, was over-cured and resulted in very low tear strengths. However, the extrusion and Gehman low temperature properties for the control were good indicating that this compound (R207721), if cured properly, should be a good final hose building formulation.

Hoses built previously from PNF^R have shown rather poor adhesion to rayon fabric. Also, one of the hoses

developed blisters on the outer cover during fueling operations. A study was undertaken to determine if the adhesion to rayon could be improved. Table XV summarizes the results of this study performed on the 33 P.H.R. FEF formulations (R207704) and different types of rayon. All attempts to bond the PNF^R-LT compound to the new types of rayon supplied by Avtex Fibers Inc. resulted in poorer adhesion than obtained with our control (Beaunit rayon with no treatment). With Beaunit rayon, dipping of the fabric in a 20% cement of compound R207704 in acetone resulted in a slight improvement in T-adhesion. Utilizing a higher peroxide level (1.2 P.H.R.) in the cement compound produced no gain in adhesion. Use of the coagent Saret-500 in the cement compound increased the T-adhesion from 10.0 to 14.0 lbs/in. A further increase (to 15.5 lbs/in) was realized through use of Silane A-174 in the cement compound. Dipping of the cord in a 10% solution of Silane A-174 in hexane resulted in the highest T-adhesion (16.5 lbs/in).

9. Hose Building Trial

A trial hose fabrication was scheduled in order to determine if the blistering observed in an earlier prepared

PNF^R-LT hose could be avoided. Two major changes were made: The cement compound included Silane A-174 as an adhesive promoter and the amount of fabric was reduced in order to permit greater strike-through of the rubber compound. The latter change would also give the hose greater flexibility. In addition, greater care was taken to avoid pick up of any foreign material during hose construction. We also tried to avoid any tearing during calendering which would require patching during hose fabrication.

Only 10-15 feet of collapsible hose were to be built in this trial effort. This small quantity precluded the use of extruded tubes and necessitated the calendering of tube and cover stocks. The compound selected was the 33 P.H.R. FEF formulation. A peroxide level study, shown in Table XVI, dictated the use of 0.9 P.H.R. of Vulcup 40KE in the tube compound and 0.8 P.H.R. in the cover stock. Relatively high cure states were attained at these levels of peroxide, but this was desired since a loss in cure state is usually observed after calendering.

The formulations used in the trial run are shown in Table XVII (R207732 and R207733) along with stress-strain data obtained before and after calendering. A one foot section of 2" hose was prepared in the lab by hand-wrapping of calendered sheets and rayon cord. This hose was then steam-cured 75 min at 320°F. As usual, a loss in tensile strength and modulus was observed after calendering (Table XVII). The stress-strain properties on the steam-cured hose were essentially the same as those obtained on the press-cured calendered stocks.

The calendering of stocks R207732 and R207733 went very well. 12-13 feet of tube, layer and cover were obtained with no tearing observed. The 0.014" layer was prepared from cover stock. The hose building also proceeded very smoothly with no difficulties encountered. A section of the hose was then tested at the Ft. Belvoir laboratories to determine if blistering would occur. After two weeks, no blisters had developed, and the hose was proclaimed satisfactory. We proceeded to the manufacture of the large lengths of hoses.

10. Final Hose Production

The compounds chosen for our final production efforts are shown in Table XVIII. The FEF black was selected over Shawinigan black because of its better low temperature properties. The peroxide level was chosen on the basis of results in the just completed trial run. A cure check on these compounds indicated properties a little lower than desired but as good as could be expected for these extrudable compounds. The cure check on the cover stocks was performed after calendering.

Our objective in this final production effort was to make 125 feet of collapsible and 35 feet of suction hoses. The tube sections for all hoses were to be extruded. The cover and layer sections were calendered sheets of cover compound R207736 which contained 2.0 parts of polybutadiene (to attain the desired difference in fuel diffusion rate of tube and cover). The calendering of both covers and the layer of the suction hose went quite well. However, considerable difficulty was encountered in calendering of the layer for the collapsible hose. The layer is a very thin sheet (0.014") which is applied between the two braids of fabric. The basic problem was the ease of tearing of this

thin sheet. Finally, we were forced to manually remove the sheets from the calender rolls, and this resulted in some folds in the rolled stock. We did manage to calender a sufficient amount of the layer but it was not in a continuous piece.

The extrusion of tube stock proceeded extremely well. A total 150 feet of collapsible tube and 50 feet of suction hose were extruded. No difficulties were encountered in attaining the desired dimensions, and the surfaces of the tubes were good.

The building of the collapsible hose proceeded as follows:

1. The extruded tubes (0.074") were lightly coated on the inside with talc following the extrusion process.
2. The extruded tubes were expanded slightly with low air pressure, and the 50 ft mandrel was pushed into the tube.
3. The tube surface was freshened with MEK and then passed through a 48 carrier horizontal braider where one layer of rayon (2200 denier, 1 ply) was applied in a 2 over, 2 under pattern (braid angle-54°).

4. The fabric-tube assembly was painted completely with a cement consisting of 20% cover stock (XS from calendering + 1.5 P.H.R. Silane A-174) dissolved in acetone.
5. The inner ply (0.014") was applied.
6. The entire assembly was passed through the braider again for application of the second layer of rayon (same as the first).
7. The fabric was painted with cement.
8. Two plies of calendered cover stock (0.037") were added to complete the hose.
9. The hose was double wrapped with wet nylon tape and cured in a steam autoclave at 320°F for 75 min. The mandrel was hollow to allow for circulation of steam.

The suction hose was built by the following procedure:

1. The extruded tube (0.10") was lightly coated on the inside with talc.
2. The 50 ft mandrel was pushed through the extruded tube which was expanded slightly with low air pressure.

3. After being freshened on the surface with MEK, the tube was passed through a 48 carrier textile horizontal braider where the first layer of rayon (2200 denier, one ply) was applied in a 2 over, 2 under pattern.
4. The fabric was completely coated with a cement of PNF^R-LT stock (same as described for the collapsible hose) in acetone.
5. One inner ply (0.037") was applied.
6. Steel wire (0.065" O.D.) was spiraled on at a spacing of 0.25".
7. Another inner ply (0.037") was applied.
8. The entire assembly was passed through the braider for application of the second layer of rayon (same as first).
9. The fabric was coated with the PNF^R-LT cement.
10. Two plies of cover stock (0.050") were added to complete the hose.
11. The hose was double wrapped with wet nylon curing tape and cured in a steam autoclave at 320°F for 75 min.

Some minor difficulties arose during the hose building process. The condition of the inner ply (0.014") slowed the hose building in that folds had to be ironed out and some splicing was required at the torn sections. The painting of fabric with cement, however, was by far the rate determining step in the entire process. For any future large scale production work, a better method of applying cement should be devised. Finally, in rolling the cover onto the hose, the sheets of rubber crimped beneath the rollers. This problem was alleviated by placing polyethylene over the rolls.

The remainder of the hose building proceeded extremely well. The extruded tubes were placed on the mandrels with no difficulties. Extrusion of the tubes certainly simplified the entire operation. Removal of the hoses from the mandrel was also trouble-free.

Since the extrusions proceeded so well, we were able to prepare a total of 150 ft of collapsible and 50 ft of suction hoses. Two of the 50 ft sections of collapsible hose were made as described earlier. With the other 50 ft section, however,

we experimented and made 42 ft of hose without any cement on the fabric.

Hydrostatic pressure test results are shown in Table XIX. Both the collapsible and suction hoses met all of the pressure test requirements. Inside diameters and weights were also within specifications for both hoses (Table XIX).

Stress-strain data before and after immersion in Type II fluid of TT-S-735 and in distilled water are summarized in Table XX. The normal properties were below specifications and lower than we had expected. Retentions of tensile strength, volume increases and % weight changes in Type II fluid were acceptable. Retentions of elongation at break were slightly below specifications.

Testing in distilled water at 160°F for 14 days showed retentions of tensile strength for both tube and cover to be good; retentions of elongation at break, however, were again slightly below specifications. Volume increases were surprisingly high for both tube and cover. After 42 days at 160°F, retentions of properties were acceptable for the cover but slightly below specification for tube stock.

Gehman low temperature properties were good for both tube and cover (Table XX). Most importantly, the G values

in the -70°F range were below 500 psi. The T_5 values were also in the desired range of -70°F or lower.

Other test results in Table XX show low temperature brittleness and weatherability to be acceptable. The existent gum contents on diced samples from the hose were within specifications. Running the same test on a length of hose resulted in a high unwashed value. Similar results were obtained with previously built PNF^R hoses.

No adhesion data is given because of difficulties encountered in trying to obtain a good test sample. Probably because of the greater strike-through and resultant tighter construction, it was impossible to get the desired separation between cover or tube and fabric layer. An uneven tearing of the rubber itself occurred, which made rubber to fabric adhesion measurement impossible. Hand testing indicated better adhesions than had ever been observed before.

Some excess of both tube and cover stocks remained from the production of hose. This material was press-cured and tested in the laboratory. Stress-strain measurements (Table XXI) showed higher tensile strengths and elongations than obtained on samples cut from the hose itself (Table XX). The 100% moduli, however, were reasonably close for the press-cured and hose samples. Tear strengths on the press-cured samples were higher than normally observed.

Small sections of hose were also prepared in the laboratory. Stress-strain data on samples removed from these hoses showed tensile strengths comparable to those of the press-cured stocks, but 100% moduli were significantly lower and elongations much higher than observed for the press-cured samples. This could be due to the fact that inadequate pressure was obtained from the hand wrapping of curing tape.

Aging in both Type II fluid and distilled water was also conducted on the press-cured samples (Table XXII). In general, retentions of properties were slightly better than observed with the samples cut from the hose. Volume increases in both fluids were comparable to values obtained from sections of the hose.

The fuel diffusion rates were also determined for both tube and cover compounds. The tube gave a value of 1.436 (fl. oz. ft⁻² 24 hrs.⁻¹) while the cover produced a rate of 1.720. Thus, the diffusion rate ratio of cover to tube was 1.2 and below the desired 1.3 ratio.

DISCUSSION

As mentioned earlier, the basic problem in this study was to develop a PNF^R-LT compound which could be extruded and yet maintained other key requirements for manufacture of a low temperature fuel hose. These requirements are:

1. Good low temperature flexibility, e.g. - Gehman T₅ of ca. -70°F and a G value of 500 psi.
2. Must be calenderable, which means the compounds must release well from mill rolls and have sufficient green strength to resist damage to the stock.
3. Must resist pull down of fabric and wire reinforcement, which again dictates good green strength.
4. Must have good adhesion - rubber and reinforcing fabric must adhere well. With the braid of rayon used, there is considerable strike-through of rubber which makes tear strength of the rubber another important feature.

5. Cured hose must have reasonable strength to withstand normal wear and tear; thus, high modulus, tensile and tear strength are desirable.
6. Good release of hose from the mandrel which should be accomplished through use of mandrel lubricants.

The previous hose prepared under contract DAAG53-75-C-0187 met these requirements (although higher tear and tensile strengths would have been desirable). Thus, our initial efforts were to investigate the FEF compound used in this previous work.

Our total batch of rubber required to meet the contract requirements was not available at the start of this investigation. Hence, some of the preliminary compounding work was done on the same blend of PNF^R-LT and PNF^R-200 used in the earlier contract effort. This blend of polymer in the 30 P.H.R. FEF formulation extruded very poorly. Increasing the black level to 40 P.H.R. produced excellent extrudates, but low temperature flexibility became unacceptable. Combinations of FEF with large particle size black or silica,

which should not have as great an effect on low temperature flexibility, gave only slight improvements in extrudability.

To improve low temperature properties, a 35 P.H.R. FEF formulation was evaluated and found to be close to low temperature flexibility specifications. However, the extrusions of this compound were not quite satisfactory.

Another black, an acetylenic type called Shawinigan, provided good extrusions at the 35 P.H.R. level, but low temperature properties were slightly poorer than observed with the 35 P.H.R. FEF stock. This compound appeared to be totally satisfactory if a small sacrifice in low temperature flexibility were acceptable.

When our total lot of polymer was available, we immediately evaluated the two best compounds (35 P.H.R. Shawinigan and 35 P.H.R. FEF). With this lot of polymer, fluorine contents were in the desired range and blending with PNF^R-200 was unnecessary. The Shawinigan and FEF compounds would not band on a mill, and it was obvious that the polymer had too much nerve to be extruded properly. This problem had been observed before with PNF^R, and heat treatment was found to be a good remedy. Consequently,

several heat-aged polymers were examined. Reasonably good extrudates were obtained for polymers aged between 6-12 hrs at 300°F. Even the FEF stocks were now acceptable with these heat aged polymers. The longer heat aging did appear to result in moderate reductions in modulus and tensile strengths. The best balance of extrudability and stress-strain properties was attainable with polymer aged for 8.5 hrs at 300°F.

Although the extrusions with 35 P.H.R. FEF were acceptable, the Shawinigan compound still appeared slightly better. However, primarily on the basis of better low temperature flexibility, we selected the FEF stock over the Shawinigan. With the FEF, acceptable extrusions were possible at the 33 P.H.R. level of black, and this compound gave better and satisfactory low temperature properties.

Having attained a suitable extrusion compound, we then proceeded to make modifications in the FEF stock in attempts to improve tear and tensile strengths. Tear strength was given much attention because the fabric braid in the hoses is such that significant strike-through of rubber occurs. Thus,

a high tear strength would have great influence on the "adhesion" of rubber to fabric. One modified compound (containing FEF, Quso WR-82, Silane A-174 and Teflon-6) gave significantly higher tear strengths. Unfortunately, this stock also showed unacceptably high Gehman T_5 and G values. Slight improvements in tear appeared possible by the addition of glass fibers and Silane A-174. However, the glass fibers had an adverse effect on extrudability. No other modified compounds gave the desired balance of properties that could be achieved with the simple FEF formulation.

In addition to working on tear strength improvements, we also tried to increase the actual adhesion of rubber to rayon. Different types of rayon as well as different treatments or dips of the rayon cord were examined. Best results (modest improvements in T-adhesions) were obtained by treating of the cord with a solution of the coupling agent Silane A-174. Either the Silane alone or as a minor component (1.5 P.H.R.) in our basic formulation was effective. The solution of the compound with added Silane was preferred, because it was felt that the added rubber compound would

provide better building tack than the Silane compound by itself.

While working on the present contract, the PNF^R-LT hose built under an earlier contract (DAAG53-75-C-0187, P00002) developed blisters on the cover during some test operations. Because of this problem, an unanticipated trial preparation of hose was scheduled. This trial run had a dual purpose: to determine if a hose could be prepared which did not develop blisters and to check out the compound proposed for use in our final hose building effort under this contract. Personnel at the Ft. Belvoir U.S. Army Mobility Equipment Research and Development Command Laboratories felt that they could run a laboratory test to determine if blistering would be a problem.

The compound selected for trial preparation is shown below:

Polymer K19166-A8.5*	-	100
FEF	-	33
MgO	-	6
Stabilizer	-	2
Vulcup 40KE	-	0.8-0.9

For tube stock, the above formulation with 0.9 P.H.R. of Vulcup 40KE was used. In the cover compound, 0.8 P.H.R.

*Polymer K19166 which was aged at 350°F for 8.5 hrs.

of Vulcup 40KE was used along with 2.0 P.H.R. of polybutadiene. The basic formulation provided the best overall balance of properties for an extrudable compound.

The blistering problem could have been caused by a variety of factors, but it was felt that improvements in "adhesion" of rubber to fabric or the bonding of tube to cover sections would certainly help in elimination of this problem. Consequently, the cement stock used in this hose building contained the Silane A-174 which seemed to provide improvements in adhesion. Furthermore, a change in fabric content was made by utilization of single rayon cord (one ply) in the braider instead of the double cords used in previous hose buildings. This change permitted still greater strike-through of rubber.

In the trial fabrication, greater precautions were taken to avoid any incorporation of foreign material during construction. The blistered hose was carefully dissected, and it appeared that foreign particles were present in a straight line under the blisters. This could have been caused by placing the wet cement-coated hose on a dirty rest pad. Greater care was also taken in the freshening operation, where MEK is brushed on the rubber surface during the cover application.

This trial preparation involved only 10-15 feet of collapsible hose which precluded the use of the large factory extruder. Consequently, the hose was made by hand wrapping of calendered tube and cover stocks. The hose building, including the calendering, progressed very smoothly. Crude examination and dissection of a piece of the resulting hose indicated better "adhesion" than ever observed previously. The reduction in amount of fabric seemed most beneficial. The hose was tested at Ft. Belvoir for a two week period, and no blisters developed.

On the basis of results from the trial run, we proceeded to manufacture of the large lengths of hose with the same FEF formulations. Only minor change was the utilization of the same level (0.9 P.H.R.) of Vulcup 40KE in both tube and cover stocks. In the trial hose, the cover showed lower modulus and tensile strength than the tube.

The manufacturing of the hoses was relatively trouble-free. The major problem encountered was the calendering of the very thin layer or inner ply (0.014") for the collapsible

hose. Calendering of this material was very difficult due to frequent tearing of the stock. This resulted in non-continuous sheets of layer which in turn caused a slow down in production of hose (had to stop to splice inner layer).

Otherwise, the hose building proceeded very well. The extrusion process in particular was a bright spot in the operation. About 200 ft of smooth-surfaced tubes were produced. These tubes possessed sufficient green strength so that they could be fitted on the mandrels with ease. This operation is certainly much simpler than hand wrapping of calendered sheets. The braiding, wire-wrapping and removal from mandrel also proceeded smoothly, and the hoses appeared to cure satisfactorily.

Another delaying factor in the manufacturing operation was the hand painting of cement on all fabric layers. This operation would definitely have to be expedited or eliminated in any larger scale productions. Since we felt the modification in fabric content was the major factor in improving ply to ply adhesions, we decided to prepare one length of collapsible hose with no cements added to fabric. Thus, two 50 ft sections of hose were made with cement and one 25 ft section was not cemented. Hopefully, tests and applications

of the two hoses will determine if cementing is necessary.

In examining the finished hoses, it also appeared that there may have been some pull down of fabric resulting in a thinner tube section than desired. This could be easily remedied by using a slightly thicker tube section with a similar reduction in thickness of cover.

With the reductions in fabric content, we were concerned about the physical strength of the hoses. Hydrostatic pressure tests indicated that some losses in strength occurred, but all requirements were still met with no difficulties.

The other major requirement of the hoses was good low temperature flexibility. Gehman testing indicated that both the tube and cover sections possessed the desired flexibility at -70°F . Both tube and cover also passed the brittleness test at -70°F .

Resistance to Type II fluid appeared to be adequate. Water resistance in terms of retention of tensile strength was adequate, but elongations for the tube stock decreased more than desired after aging at 160°F and volume increases did not meet the specifications. Earlier studies had shown no problems in distilled water with the $\text{PNF}^{\text{R}}\text{-LT}$, so the results were surprising and perplexing.

Normal stress-strain properties measured on samples taken from the hose were quite low relative to specifications. One cause of the low tensile strength may be the presence of defects in samples obtained from the hose. The strips of tube and cover are quite thin to begin with, and this makes it extremely difficult to buff the samples to a smooth surface. If defects were present, lower tensile strengths and elongations should be observed; modulus at low strain could remain unchanged. Actually, in comparing stress-strain of samples from hose vs. press-cured samples, this is what is observed. The 100% moduli were essentially comparable while tensile strengths and elongations were lower for the samples taken from the hoses. Tensile strengths for the press-cured samples were still below specifications but probably as high as could be expected for compounds which could be extruded and maintained -70°F flexibility.

After accelerated weathering, the cover stock showed essentially no change in stress-strain properties. Ozone resistance for the cover was also good.

The existent gum tests again gave us differing results dependent on whether the hose itself or diced samples were tested. The diced sample showed acceptable results whereas the hose itself gave a particularly high unwashed value. The residues were not examined.

Adhesion data could not be obtained on the hoses. In order to test adhesions of tube and cover to fabric, it is necessary to take a 1" section of hose and neatly pull apart the tube or cover away from the fabric. With the reduced amount of fabric and greater strike-through of rubber, this clean separation of rubber from fabric was not attainable. Instead, the rubber phase would tear resulting in less than the desired 1" surface and invalid results. One reasonable test was obtained for cover to fabric on an uncemented hose section. This test gave an adhesion value of 12 lbs/in. Repeated tests on the other hoses failed to give satisfactory tests. Hand pulling indicated that adhesions were better than ever observed previously. Field testing will probably give better indications of the bonding or adhesion in the hoses. Hopefully these field tests will also provide some comparisons of the cemented vs uncemented hoses.

The tear strengths observed for the final compounds were slightly higher than normally observed. However, further improvements would certainly be desired. The tearing of the rubber during attempted adhesion tests attests this need.

Overall, the results of large scale production of PNF^R-LT Arctic fuel hoses via the extrusion process were quite satisfying. Major objectives were realized and adequate amounts of hoses with good low temperature flexibility and dimensional stability were produced.

CONCLUSIONS

It has been demonstrated that a PNF^R-LT compound can be extruded in a factory operation to produce suitable tube sections for both collapsible and suction type fuel hoses. Large lengths of both types of hoses were fabricated without any major difficulties.

Laboratory test results on the hoses were in general fairly good. Dimensional stability and physical strength as determined by hydrostatic pressure testing were good for both types of hoses. On the basis of Gehman and brittleness tests, the critical low temperature flexibility of these hoses should be good. Retentions of stress-strain properties in Type II Fluid and in water (160°F) were close to specifications, but volume swell in water was unusually high. Weatherability of the cover was acceptable. As observed in earlier-prepared PNF^R hoses, normal tensile strength was below desired specifications. Again, more reinforcing fillers which could improve tensile strength were avoided in order to attain good low temperature flexibility and extrudability.

It was also demonstrated that these hoses could tolerate significantly lower amounts of fabric. With the reduced fabric, greater strike-through of rubber was attained and this appeared to yield a better bond between tube-ply-cover sections. The reduction in fabric content also yielded a more flexible hose.

Sufficient lengths of hoses were produced for actual field testing in the Arctic. Important conclusions regarding the performance capabilities of these hoses should be forthcoming from such tests.

RECOMMENDATIONS

Although major objectives of this investigation were achieved, some minor problems still require attention. In the manufacturing process, calendering of the thin layer for collapsible hose, applications of cement and pull down of cord were areas of concern. The thickness of the layer could probably be increased from 0.014" to ca. 0.025" without greatly affecting the weight of hose. This thicker sheet should calender readily. The cord pull

down problem could also be alleviated by a change in dimensions of tube (to thicker sheet) and cover (to thinner sheet). The cement application could be facilitated by treating the cord with cement. Hopefully, field trials on the uncemented hose will show if the cement can be simply eliminated.

Tensile and tear strengths still appear to be problems. A compromise in low temperature properties would improve this situation. Also, if non-extractable oils or plasticizers were available, it may be possible to achieve higher tensile strengths with a combination of the higher molecular weight polymer (unaged) and oils or plasticizers. It may also be possible that field tests will indicate acceptable performance and no need for higher tear or tensile strengths.

GLOSSARY

PNF ^R -200	A phosphonitrilic fluoroelastomer containing pendant fluoroalkoxy groups and supplied by Firestone.
PNF ^R -LT	A modification of PNF ^R -200 in which the level of fluorine in the polymer is reduced.
Polybutadiene	HD-35, a 35 Mooney viscosity polymer supplied by Firestone.
MgO	Stan Mag ELC supplied by Harwick (Akron, Ohio).
Stabilizer	Zinc II bis(8-oxyquinolate).
Vulcup 40KE	40% α, α' -bis(t-butylperoxy)diisopropylbenzene dispersed on Burgess 40KE. Supplied by Hercules.
Shawinigan Black	A black made from acetylene gas and supplied by Gulf Oil Canada Limited.
Teflon 8A	A fibrous Teflon supplied by Dupont.
Teflon 6	A powdered Teflon supplied by Dupont.
P.H.R.	Parts per hundred rubber.
FEF	Fast extruding furnace black with ASTM classification N550.
Austin Black	Finely ground coal made by Slab Fork Coal Co.
Quso WR-82	A silane-treated, precipitated silica supplied by Philadelphia Quartz (Valley Forge, PA)

GLOSSARY (Cont.)

Silane A-174	Gamma-methacryloxypropyltrimethoxyl silane supplied by Union Carbide Co.
Saret 500	Cross-linking agent supplied by Sartomer Co. (West Chester, PA)
Imsil A-108 (A-174)	An amorphous silica (Grade A-108) treated with 1 wt % Silane A-174 and provided by Illinois Minerals Co.
Imsil A-108 (A-1100)	An amorphous silica (Grade A-108) treated with 1 wt % Silane A-1100 and provided by Illinois Minerals Co.
Imsil A-10 (A-1100)	An amorphous silica (Grade A-10) treated with 1 wt % Silane A-1100 and provided by Illinois Minerals Co.
Beaunit Rayon	Rayon cord used at American Biltrite in final hose fabrication; supplied by Beaunit Co.
Avtex Rayons	Rayon fibers with proprietary treatments provided by Avtex Fibers Inc. (Valley Forge, PA).
Glass Fiber	Fibers (W3080M) obtainable from PPG; these fibers were also treated with a coupling agent supplied by Dow Corning (Z6080).

TABLE I
EXTRUSION PROFILE FOR FEF COMPOUND

<u>Stock</u>	<u>R203835</u>
PNF ^(R) -LT (K15900)	60.0
PNF ^(R) -200 (RPP10424)	40.0
FEF	30.0
MgO	6.0
Stabilizer	2.0
Vulcup 40KE	1.1
<u>Normal Stress-Strain - cure: 35' @ 320°F</u>	
100% M, psi	948
Tensile, psi	1317
Ult. Elong., %	140
<u>Garvey Die (Method A - ASTM D2230) - Extrusion Temp. = 170°F</u>	
10 sec. length, cm	19.05
10 sec. wt., g	25.60
Swell Index, g/cm	1.34
Extrusion Rate, g/min.	153.6
Contour	1
Corners	1
Edge	1
Surface	1
<hr/>	
Total	4

TABLE II

EXTRUDABILITY OF FEF-IMSIL COMPOUNDS

<u>Stock R203</u>	<u>855</u>	<u>856</u>	<u>857</u>	<u>858</u>	<u>859</u>
PNF ^(R) -LT (K15900)	60.0	60.0	60.0	60.0	60.0
PNF ^(R) -200 (RPP10424)	40.0	40.0	40.0	40.0	40.0
FEF	20.0	20.0	10.0	20.0	20.0
Imsil A-108 (A-174)	20.0	30.0	30.0	-	-
Imsil A-108 (A-1100)	-	-	-	30.0	-
Imsil A-10 (A-1100)	-	-	-	-	30.0
MgO	6.0	6.0	6.0	6.0	6.0
Stabilizer	2.0	2.0	2.0	2.0	2.0
Vulcup 40KE	0.8	0.8	0.8	0.8	0.8
<u>Brabender Extrusion</u> - Head T°C = 100, Barrel T°C = 95, 0.25" OD die.					
rpm	60	60	60	60	60
g/min.	46	72	72	68	71
diameter, in.	0.32	0.31	0.31	0.31	0.31
rating	poor-fair fair-poor poor fair-poor poor-fair				
<u>Normal Stress-strain</u>					
100% M, psi	840	1123	874	1130	945
Tensile, psi	1148	1172	1015	1130	1070
Ult. Elong., %	135	110	120	100	120

TABLE III

EVALUATION OF FEF, FEF-AUSTIN, SHAWINIGAN BLACKS

Stock R203-	844	845	846	847
PNF (R)-LT (K15900)	60.0	60.0	60.0	60.0
PNF (R)-200 (RPP10424	40.0	40.0	40.0	40.0
FEF	30.0	30.0	40.0	-
Austin	-	20.0	-	-
Shawinigan	-	-	-	30.0

All stocks also included: 6 MgO, 2 stabilizer, 0.8 Vulcup 40KE

Mix Evaluation

Brabender mix	good	good	good	good
Dump condition	good	good	good	good
Dump time	8 min	8 min	8 min	8 min
Milling	good	fair	good	good

Brabender Extrusion - Head T°C = 100, Barrel T°C = 95, 0.25" O.D. die

R.P.M.	60	60	60	40
g./min	66	ND	56	36
Diameter, in.	0.35	0.31	0.30	0.32
Rating	poor	poor	good	fair

Normal Stress-Strain - Cure: 35' @ 320°F

100% M, psi	686	755	725	1055
Tensile, psi	1354	887	1022	1105
Ult. Elong, %	165	135	160	110

Gehman Low Temp. Properties - Cure: 40' @ 320°F

T ₅ °F	-76	-74	-66	-76
G ₅ @ R.T., psi	79.5	125.3	144.0	135.5
G @ -55°C, psi	258.0	437.5	770.7	437.5

TABLE IV
EVALUATION OF FEF AND SHAWINIGAN BLACKS AT 35 P.H.R.

<u>Stock R203</u>	<u>-862</u>	<u>-863</u>
PNF(R)-LT (K15900)	60.0	60.0
PNF(R)-200 (RPP10424)	40.0	40.0
Shawinigan	35.0	-
FEF	-	35.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	0.6	0.8
<u>Milling</u>	good	fair
<u>Brabender Extrusion</u> - Head T°C = 100, Barrel T°C = 99, 0.25" O.D. die		
r.p.m.	60	60
g./min.	48	56
diameter, in.	0.313	0.340
rating	good	poor+
<u>Normal Stress-Strain</u> - Cure: 35' @ 350°F - <u>before extrusion</u>		
100% M, psi	1166	835
Tensile, psi	1166	1168
Ult. Elong. %	110	165
<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F - <u>after extrusion</u>		
100% M, psi	738	441
Tensile, psi	1313	1200
Ult. Elong. %	150	225
<u>Trouser Tear @ R.T.</u> - Cure: 40' @ 320°F		
lbs./in.	19	25
<u>Gehman Low Temp. Properties</u> - Cure: 40' @ 320°F		
T ₅ , °F	-71.5	-72.6
G ₅ @ R.T., psi	184	117
G @ -55°C, psi	633	507

TABLE V

EFFECT OF COUPLING AGENT, GLASS FIBER IN FEF FORMULATION

<u>Stock R203</u>	<u>-836</u>	<u>-837</u>	<u>-838</u>	<u>-839</u>	<u>-840</u>
PNF ^(R) -LT (K15900)	60.0	60.0	60.0	60.0	60.0
PNF ^(R) -200 (RPP10424)	40.0	40.0	40.0	40.0	40.0
FEF	30.0	30.0	30.0	30.0	30.0
MgO	6.0	6.0	6.0	6.0	6.0
Stabilizer	2.0	2.0	2.0	2.0	2.0
Treated Fiber*	-	-	-	2.0	2.0
Silane A-174	-	1.5	-	1.5	-
Untreated Fiber*	-	-	2.0	-	-
Vulcup 40KE	1.1	0.8	1.0	0.8	1.0

Normal Stress-Strain - Cure: 35' @ 320°F

100% M, psi	1100	1214	1076	-	-
Tensile, psi	1100	1214	1198	1082	1224
Ult. Elong., %	100	100	110	80	85

The peroxide and silane levels on the above stocks were adjusted and resubmitted as follows:

<u>Stock R203</u>	<u>-850</u>	<u>-851</u>	<u>-852</u>	<u>-853</u>
Silane A-174	-	1.2	-	1.0
Vulcup 40KE	0.8	0.6	0.8	0.5

Milling good good good good

Normal Stress-Strain - Cure: 35' @ 320°F

100% M, psi	697	792	607	871
Tensile, psi	1106	1183	879	1065
Ult. Elong., %	170	155	155	120

Trouser Tear @ R.T. - Cure: 40' @ 320°F

lbs./in.	12	19	14	26
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Gehman Low Temp. Properties - Cure: 40' @ 320°F

T ₅ , °F	-72.4	-70.0	-74.6	-75.6
G @ R.T., psi	113	101	155	80
G @ -55°C, psi	233	448	234	316

*See Glossary

TABLE VI

EFFECT OF TEFLON-6, SILANE A-174 AND QUSO WR82

<u>Stock</u>	<u>R203849</u>
PNF ^(R) -LT (K15900)	60.0
PNF ^(R) -200 (RPF10424)	40.0
FEF	23.0
Quso WR-82	12.0
Teflon-6	3.0
Silane A-174	1.0
MgO	6.0
Stabilizer	2.0
Vulcup 40KE	0.6
<u>Brabender Extrusion - Head T °C = 100, Barrel T °C = 90,</u> 0.25" OD die	
r.p.m.	60
g./min.	55
diameter, in.	0.334
rating	poor +
<u>Normal Stress-Strain - Cure: 35' @ 320°F - before extrusion</u>	
100% M, psi	1069
Tensile, psi	1211
Ult. Elong., %	140
<u>Normal Stress-Strain - Cure: 35' @ 320 °F - after extrusion</u>	
100% M, psi	916
Tensile, psi	1160
Ult. Elong., %	160
<u>Trouser Tear @ R.T. - Cure: 40' @ 320°F</u>	
lbs./in.	60
<u>Gehman Low Temp. Properties - Cure: 40' @ 320°F</u>	
T ₅ , °F	-56.0
G ₅ @ -55°C, psi	1821
G @ R.T., psi	340

TABLE VII

EVALUATION OF SHAWINIGAN BLACK-TEFLON 8-A

<u>Stock R203</u>	<u>-860</u>	<u>-861</u>
PNF ^(R) -LT (K15900)	60.0	60.0
PNF ^(R) -200 (RPP10424)	40.0	40.0
Shawinigan	35.0	35.0
Teflon 8-A	1.0	2.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	0.6	0.6

Mix Evaluation

Brabender Mix	good	good
Dump Condition	good	good
Dump T, °F	285	350
Milling	good	good

Brabender Extrusion - Head T °C = 100, Barrel T °C = 95, 0.25" O.D.

r.p.b.	60	60
g./min.	56	53
diameter, in.	0.329	0.339
rating	fair-	poor+

Normal Stress-Strain - Cure: 35' @ 320°F - before extrusion

100% M, psi	1190	1261
Tensile, psi	1217	1319
Ult. Elong, %	105(R)	110

Normal Stress-Strain

100% M, psi	858	829
Tensile, psi	1290	1200
Ult. Elong, %	150	155

Trouser Tear @ R.T. - Cure: 40' @ 320°F

lbs./in.	17	26
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Gehman Low Temp. Properties - Cure: 40' @ 320°F

T ₅ , °F	-59.1	-57.8
G ₅ @ R.T., psi	163	157
G @ -55°C, psi	945	1694

TABLE VIII

PROPERTIES OF INDIVIDUAL POLYMERS

	<u>RFP No.</u>	<u>DSV</u>	<u>% Gel</u>	<u>Wt.% F⁽¹⁾</u>	<u>Tg °C</u>	<u>% Vol. ⁽²⁾</u>	<u>T₅ °F⁽³⁾</u>	<u>G(-55°C)</u>
1.	10892	2.36	0.0	49.6	-73	15.5	-67.2	272
2.	10893	2.42	0.0	49.1	-74	15.7	-67.0	286
3.	10900	2.39	0.0	43.5	-78.5	27.6	-66.8	201
4.	10901	2.75	0.0	47.7	-78.5	29.2	-68.3	184
5.	10902	3.07	0.0	47.3	-78.0	26.2	-75.2	255
6.	10915	3.37	0.0	45.7	-81.0	45.5		
7.	10920	3.25	0.0	47.8	-80.5	41.4		
8.	10926	2.56	0.0		-78.0			
9.	10927	2.82	0.0		-78.5		-75.6	358
10.	10928	2.55	0.0		-80.0			
11.	9848	3.07	0.0		-81.0	43.8		
12.	10206	3.94	0.0	46.5	-79.0			
13.	10207	3.98	0.0	44.3	-79.0			
14.	10208	3.39	0.0	48.1	-79.5			

(1) Calculated value, based on NMR analysis of pendant group ratio.

(2) After 15 days (R.T.) in Type II Fluid (TT-S-735).

(3) Gehman data was obtained on standard formulation (30 FEF, 6 MgO, 2 Stabilizer, 0.7 Vulcup 40KE).

TABLE IX

PROPERTIES OF BLEND OF POLYMERS, OPTIMIZATION OF HEAT AGING TIME

K19166 = a blend of 14 polymers listed in Table V

<u>Sample No.</u>	<u>Hrs. @ 300°F</u>	<u>D.S.V.</u>	<u>% Gel</u>		
K19166	0	2.10	0.0		
K19166-A4	4	1.66	0.0		
K19166-A6	6	1.55	0.0		
K19166-A8	8	1.00	0.0		
K19166-A10	10	1.05	0.0		
K19166-A12.5	12.5	0.91	0.0		
<u>Stock R203</u>	<u>-888</u>	<u>-889</u>	<u>-890</u>	<u>-893</u>	<u>-894</u>
Polymer	K19166-A4	K19166-A6	K19166-A8	K19166-A10	K19166-A12.5
Formulation -	all contained 30 FEF, 6 MgO, 2 Stabilizer, 0.7 Vulcup 40KE				
<u>Milling</u>	good	good	good	good	good+
<u>Extrusion</u> - Brabender, 0.25" die, Head T°C = 100, Barrel T°C = 89-95, 60 rpm					
pressure, psi	1500	1500	800	900	700
g./min.	66	53	28	53	32
diameter, in.	0.320	0.312	0.305	0.300	0.280
rating	poor	fair-	fair+	fair+	good-
<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F, <u>before extrusion</u>					
100% M, psi	923	936 ^(R)	844	940	782
Tensile, psi	1371	1282	1144	1130	1048
Ult. Elong., %	135	130	125	115	130
<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F, <u>after extrusion</u>					
100% M, psi	765	825	858	875	870
Tensile, psi	1283	1220	1152	1045	1110
Ult. Elong. %	135	135	130	115	125
<u>Trouser Tear</u> @ R.T. - Cure: 40' @ 320°F					
lbs./in.	10.0	7.0	10.0	12.0	13.0
<u>Gehman Low Temp. Properties</u> - Cure: 40' @ 320°F					
T ₅ ^{°F}	-70.8	-67.2	-70.8	-66.3	-65.9
G ₅ @ R.T., psi	101	77	85	86	84
G @ -55°C, psi	399	394	407	512	479

TABLE X

EVALUATION OF 35 p.h.r. FEF AND SHAWINIGAN BLACK COMPOUNDS

<u>Stock R203</u>	<u>-891</u>	<u>-892</u>
Polymer	K19166-A8	K19166-A8
Black	FEF	Shawinigan
Remaining formulation: 6 MgO, 2 Stabilizer, 0.7 Vulcup 40KE		
<u>Milling</u>	good+	good+
<u>Extrusion</u> - Brabender, 0.25" die, Head T°C = 100, Barrel T°C=91		
pressure, psi	900	1650
g./min.	16	48
diameter, in.	0.290	0.282
rating	good	good+
r.p.m.	60	60
<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F, <u>before extrusion</u>		
100% M, psi	981	-
Tensile, psi	1196	1107
Ult. Elong. %	107	80
<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F, <u>after extrusion</u>		
100% M, psi	920	-
Tensile, psi	1220	1155
Ult. Elong, %	130	80
<u>Trouser Tear @ R.T.</u> - Cure: 40' @ 320°F		
lbs./in.	20	16
<u>Gehman Low Temp. Properties</u> - Cure: 40' @ 320°F		
T ₅ , °F	-63.2	-60.9
G ⁵ @ R.T., psi	139	175.9
G @ -55°C, psi	897	1180

TABLE XI

COMPARISON OF 35 AND 33 P.H.R. FEF STOCKS

<u>Stock R207-</u>	<u>700</u>	<u>701</u>	<u>702</u>
Polymer (100 parts)	K19166-A6	K19166-A7.5	K19166-A7.5
FEF	35	35	33
Remaining formulation for all stocks: 6 MgO, 2 stabilizer, 0.6 Volcup 40KE			
<u>Milling</u>	good+	good+	good
<u>Brabender Extrusion</u> - Head T. ^{°C.} = 100, Barrel T. ^{°C.} = 95, r.p.m. = 60, 0.25" die			
pressure, psi	1150	1450	1350
g./min.	52	64	58
diameter, in.	0.297	0.298	0.301
rating	fair+	fair+	fair+
<u>Normal Stress-Strain</u> - cure: 35' @ 320 [°] F - <u>before extrusion</u>			
100% M, psi	772	786	837
Tensile, psi	1081	1098	1130
Ult. Elong., %	140	145	135
<u>Normal Stress-Strain</u> - cure: 35' @ 320 [°] F - <u>after extrusion</u>			
100% M, psi	785	800	713
Tensile, psi	1091	1047	1131
Ult. Elong. %	140	130	170
<u>Trouser Tear @ R.T.</u> - cure: 40' @ 320 [°] F			
lbs./in.	9	9	9
<u>Gehman Low Temp. Properties</u> - cure: 40' @ 320 [°] F			
T ₅ , [°] F	-74.9	-75.6	-85.4
G ² @ R.T., psi	134	125	125
G @ -55 [°] C, psi	454	507	325

TABLE XII
EFFECT OF SILANE A-174 IN BLACK AND
BLACK-SILICA FORMULATIONS

<u>Stock R207-</u>	<u>708</u>	<u>709</u>
Polymer (K19166-A7.5)	100.0	100.0
FEF	23.0	33.0
Quso WR-82	12.0	-
Teflon -6	1.0	-
Silane A-174	1.0	1.5

Remaining formulation for both stocks: 6 MgO, 2 stabilizer, 0.6 Vulcup 40KE

<u>Milling</u>	good	good
<u>Brabender Extrusion</u> - Head T °C = 96, r.p.m. = 60, 0.25" die		
pressure, psi	1250	1150
g./min.	72	54
diameter, in.	0.300	0.299
rating	fair	fair

<u>Normal Stress-Strain</u> - Cure: 35' @ 320°F.		
100% M, psi	833	925
Tensile, psi	1012	1100
Ult. Elong., %	120	120

<u>Trouser Tear @ R.T.</u>		
lbs./in.	10	9

Gehman Low Temp. Properties

T ₅ , °F	-59.3	-59.4
G ² @ R.T., psi	160	136
G @ -55°C, psi	960	796

<u>T-Adhesion @ R.T.</u>		
lbs./in.	8.0	6.5

TABLE XIII
EVALUATION OF GLASS FIBER IN FEF STOCK

<u>Stock R207-</u>	<u>724</u>	<u>725</u>
Silane A-174	1.5	1.5
Treated fiber*	2.0	-
Untreated fiber*	-	2.0
Vulcup 40KE	0.6	0.5

Remaining formulations: 100 polymer, 6 MgO, 2 stabilizer, 33 FEF

<u>Milling</u>	good	good
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Brabender Extrusion - Head T°C = 96, r.p.m. = 60, 0.25" die

pressure, psi	1250	1450
g./min.	62	59
diameter, in.	0.298	0.296
rating	fair	fair

Normal Stress-Strain - Cure: 35' @ 320°F, after extrusion

100% M, psi	850	825
Tensile, psi	1000	1050
Ult. Elong., %	120	120

Trouser Tear @ R.T. - Cure: 40' @ 320°F

lbs./in.	13	11
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Gehman Low Temp. Properties - Cure: 40' @ 320°F

T ₅ , °F	-72.4	-74.2
G ⁵ @ R.T., psi	141	133
G @ -55°C, psi	652	467

T-Adhesion @ R.T.

lbs./in.	7.0	7.5
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*See Glossary

TABLE XIV
EVALUATION OF COAGENT (SARET 500) AND
SAF IMSIL COMBINATIONS

<u>Stock R207-</u>	<u>719</u>	<u>720</u>	<u>717</u>	<u>721</u>
Polymer (K19166-A.5)	100.0	100.0	100.0	100.0
SAF	-	30.0	20.0	-
Imsil A-10 (A-1100)	-	20.0	30.0	-
FEF	33.0	-	-	33.0
Saret-500	1.5	-	-	-
Vulcup 40KE	0.6	1.1	1.0	0.8

Remainder of each formulation: 6 MgO, 2 stabilizer

<u>Milling</u>	good	poor	poor	good
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Brabender Extrusion -- Head T. °C. = 100, Barrel T. °C. = 95,
r.p.m. = 100, 0.25" die

pressure, psi	1050	1700	850	1100
g./min.	64	50	48	62
diameter, in.	0.284	0.294	0.294	0.289
rating	good+	fair-	fair-	good

Normal Stress-Strain - Cure: 35' @ 320°F, before extrusion

100% M, psi	952	730	810	-
Tensile, psi	1083	1154	1176	1350
Ult. Elong, %	120	160	140	80

Normal Stress-Strain - Cure: 35' @ 320°F, after extrusion

100% M, psi	829	700	740	-
Tensile, psi	1137	1113	1115	1337
Ult. Elong, %	135	170	140	80

Trouser Tear @ R.T. - Cure: 40' @ 320°F

lbs./in.	12	13	10	8
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Gehman Low Temp. Properties - Cure: 40' @ 320°F

T ₅ , °F	-66.6	-55.3	-78.7	-78.7
G @ R.T., psi	145	180	240	139
G @ -55°C., psi	710	1235	648	512

TABLE XV
ADHESION STUDIES

Stock - R207704

Formulation - 100 polymer (K19166-A7.5), 33 FEF,
6 MgO, 2 stabilizer, 0.7 Vulcup 40KE

Normal Stress-Strain - Cure: 35' @ 320°F

100% M, psi 785
Tensile, psi 1154
Ult. Elong, % 150

T - Adhesion

	<u>Cord/treatment</u>	<u>lbs./in.</u>
1.	Beaunit rayon/none	10.0
2.	Beaunit rayon/dipped in 20% R207704 cement	11.5
3.	Beaunit rayon/dipped in 20% R207770YA (high peroxide) cement	9.8
4.	Beaunit rayon/dipped in 20% R207719 (coagent-Table XIV) cement	14.0
5.	Beaunit rayon/dipped in 20% R207724 (Silane-Table XIII) cement	15.5
6.	Beaunit rayone/dipped in 10% Silane A-174 in hexane	16.5
7.	Avtex 25 rayon/none	5.5
8.	Avtex 25 rayon/dipped in 20% R207704 cement	6.5
9.	Avtex 57 rayon/none	6.5
10.	Avtex 57 rayon/dipped in 20% R207704 cement	7.5
11.	Avtex 37 rayon/none	7.5
12.	Avtex 37 rayon/dipped in 20% R207704 cement	9.0

All cements were made by dissolving stocks in acetone.

TABLE XVI
DETERMINATION OF CURATIVE LEVEL FOR
TRIAL HOSE BUILDING

<u>Stock R207-</u>	<u>727</u>	<u>728</u>	<u>729</u>
Polymer (K19166-A8.5)	100.0	100.0	100.0
FEF	33.0	33.0	33.0
MgO	6.0	6.0	6.0
Stabilizer	2.0	2.0	2.0
Vulcup 40KE	0.7	0.9	1.1
<u>Normal Stress-Strain</u> - press-cure: 35' @ 320°F			
100% M, psi	755	960	-
Tensile, psi	1040	1065	1090
Ult. Elong, %	130	110	90
<u>Normal Stress-Strain</u> - On Samples of Hose steam cured 90' @ 320°F			
100% M, psi	810		-
Tensile, psi	979	N.D.	880
Ult. Elong, %	115		80

TABLE XVII

PREPARATION AND CURE - CHECKING OF STOCKS
FOR TRIAL HOSE BUILDING

<u>Stock R207-</u>	<u>732 (tube)</u>	<u>733 (cover)</u>
Polymer (K19166-A8.5)	100.0	100.0
Polybutadiene	-	2.0
FEF	33.0	33.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	0.9	0.8

Normal Stress-Strain - press-cure: 320°F

100% M, psi

35'	764	655
45'	852	716

Tensile, psi

35'	968	1022
45'	1059	976

Ult. Elong, %

35'	150	175
45'	135	150

Normal Stress-Strain - on hose samples steam cured 75' @ 320°F

100% M, psi	711	532
Tensile, psi	1000	839
Ult. Elong, %	132	148

TABLE XVIII

PREPARATION AND CURE - CHECKING OF STOCKS
FOR FINAL HOSE BUILDING

<u>Stock R207</u>	<u>736 (cover)</u>	<u>737 (tube)</u>
Polymer (K19166-A8.5	100.0	100.0
Polybutadiene	2.0	-
FEF	33.0	33.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	0.9	0.9
<u>Normal Stress-Strain</u> - press-cured @ 320°F (cover - after calendering)		
<u>100% M, psi</u>		
35'	590	677
45'	655	738
<u>Tensile, psi</u>		
35'	925	979
45'	900	1015
<u>Ult. Elong, %</u>		
35'	160	135
45'	145	135

TABLE XIX

TEST RESULTS ON FINAL HOSES -
PHYSICAL REQUIREMENTS

<u>A. Collapsible Hose</u>	<u>Result</u>	<u>Spec</u>
Inside Diameter	2.03"	2 \pm 0.1"
Weight	14.5 oz/ft	16 oz/ft(max)
Hydrostatic Proof	No leaks or imperfections	No leaks or imperfections
Length Change and Twist	-0.4% 0	Length \pm 3%(max) Twist 7 $^{\circ}$ /ft(max)
Burst Pressure	375 psi	200 psi (min)
<u>B. Suction Hose</u>		
Inside Diameter	2.0625"	2 \pm 0.0625"
Weight	29.8 oz/ft	32 oz/ft (max)
Length Change and Twist	+2.5% 2 $^{\circ}$ /ft	Length +3% (max) Twist 7 $^{\circ}$ /ft (max)
Burst Pressure	500 psi	200 psi (min)
Crush Resistance -	87.4%	85% under load (max)
% of original O.D.	97.3%	95% after load release (max)

TABLE XX

PROPERTIES OF TUBE AND COVER OF FINAL HOSE

Initial Stress-Strain	Tube				Cover			
	Obtained		Spec		Obtained		Spec	
100% M, psi	674		--		538		--	
Tensile, psi	852		1500		742		1500	
Ult Elong, %	138		150		128		150	
Immersed in Type II Fluid of TT-S-735 @ R.T. for								
	Tube				Cover			
	94	14	94	14	94	14	94	14
	hrs	Spec	Days	Spec	hrs	Spec	Days	Spec
100% M retained,%	77.2	--	87.3	--	84.5	--	83.7	--
Tensile retained, %	61.0	60.0	69.0	60.0	61.3	40.0	60.8	40.0
Ult elong retained, %	72.5	85.0	72.5	80.0	78.3	80.0	74.2	75.0
Volume increase,%	29.0	40.0	29.5	40.0	25.6	70.0	32.9	70.0
Wt change, % loss	--	--	1.0	5.0	--	--	1.1	5.0
Immersed in Distilled Water @ 160°F for								
	Tube				Cover			
	14	42	14	42	14	42	14	42
	Days	Spec	Days	Spec	Days	Spec	Days	Spec
100% M retained,%	100.0	--	100.0	--	100+	--	100.0	--
Tensile retained,%	79.8	80.0	56.2	60	90.6	80.0	65.1	60
Ult Elong retained, %	68.8	80.0	41.8	60	74.2	80.0	64.7	60
Volume increase,%	39.1	15.0		20	38.2	15.0		20
After Accelerated Weathering (500 hrs)								
					Cover Only			
					Found	Spec		
Tensile retained, %					100	85		
Ult Elong retained, %					91.7	85		
After Ozone Exposure								
					Cover Only: No cracking or checking.			
Existent Gum								
					Found	Spec		
Unwashed, mg/100 ml)					630	20		
Washed, mg/100 ml)					26	5		
" mg/100 ml -on diced sple					3.6	5		
Unwashed, mg/100 ml					12.4	20		
Brittleness								
After 160 hrs @ -70°C					Pass	Pass		
Gehman Properties								
					Tube	Cover		
T ₅ °F					-68.8	-80.7		
G @ R.T., psi					88.7	148.2		
G @ -55°C, psi					413.3	495.7		

TABLE XXI

STRESS-STRAIN ON SAMPLES OF EXCESS
STOCK FROM FINAL HOSE BUILDING

<u>Stock</u>	<u>R207736 (Cover)</u>	<u>R207737 (Tube)</u>
<u>Normal Stress-Strain</u> - Press-cure: 45' @ 320°F		
100% M, psi	555	607
Tensile, psi	928	1003
Ult Elong, %	173	163
<u>Normal Stress-Strain</u> - Steam-cured hose: 75' @ 320°F		
100% M, psi	234	286
200% M, psi	557	730
Tensile, psi	875	1021
Ult Elong, %	275	250
<u>Trouser Tear @ R.T.</u> - on Press-cured Slab (45' @ 320°F)		
lbs/in	16.0	20.0

TABLE XXII
FLUID AGING ON PRESS-CURED SAMPLES OF EXCESS
STOCK FROM FINAL HOSE BUILDING

<u>Stock</u>	<u>Tube (R207737)</u>		<u>Cover (R207736)</u>	
<u>Aged 94 hrs in Type II Fluid (R.T.)</u>				
		%		%
	<u>Original</u>	<u>Retained</u>	<u>Original</u>	<u>Retained</u>
100% M	638	65.2	195	67.2
Tensile Strength	986	77.7	915	64.5
Ult Elong	93	96.8	170	94.1
Volume Increase, %	29.5		34.1	
<u>Aged 14 Days in Type II Fluid (R.T.)</u>				
100% M	782	72.6	644	60.4
Tensile Strength	989	66.7	927	70.7
Ult Elong	133	92.5	157	87.2
Volume Increase, %	32.1		36.1	
<u>Aged 14 Days in Dist. Water (160°F)</u>				
100% M	859	80.4	650	93.7
Tensile Strength	973	77.8	877	80.2
Ult Elong	120	91.7	150	80.0
Volume Increase, %	31.2		33.1	

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Phosphonitrilic Fluoroelastomer Fuel Hose -- Utilization of Extruded Tubes		5. TYPE OF REPORT & PERIOD COVERED Final report 18 August 76 - 18 July 77
7. AUTHOR(s) T. A. Antkowiak		6. PERFORMING ORG. REPORT NUMBER 1432-2
9. PERFORMING ORGANIZATION NAME AND ADDRESS Central Research Laboratories The Firestone Tire & Rubber Co. Akron, Ohio 44317		8. CONTRACT OR GRANT NUMBER(s) DAAK70-76-C-0239
11. CONTROLLING OFFICE NAME AND ADDRESS		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE October 1977
		13. NUMBER OF PAGES 1286p
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)		
<div style="border: 1px solid black; padding: 5px; text-align: center;"> DISTRIBUTION STATEMENT A Approved for public release; Distribution Unlimited </div>		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
Approved for public release; distribution unlimited.		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Phosphonitrilic fluoroelastomers (PNF ^R) Elastomers Arctic fuel hose Type II fluid resistance Low temperature fuel hose Compounding Extrusion of PNF ^R		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
This report discusses the preparation of fuel resistant fuel hoses that are serviceable at temperatures as low as -70°F. The hoses were made from a phosphonitrilic fluoroelastomer with the tube sections extruded. Work directed toward attainment of extrudable compounds, the hose building procedure and laboratory evaluations of the hoses are also described in the report.		